

PART III

EPA Region 5 Records Ctr.



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ADDENDUM 1

TO

FINAL REPORT on X-RAY FLUORESCENCE FIELD STUDY
OF SELECTED PROPERTIES IN VICINITY OF FORMER
USS LEAD REFINERY FACILITY, EAST CHICAGO, INDIANA

June 14, 2004

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Addendum I to Final Report on X-ray Fluorescence Field Study Of Selected Properties in Vicinity of Former USS Lead Refinery Facility, East Chicago, Indiana, June 14, 2004

1. INTRODUCTION

This Addendum has been prepared for the following purposes:

- To provide the results from confirmatory laboratory analysis conducted by American Analytical & Technical Services (AATS) on bulk soil samples received from USEPA via chain of custody on August 28, 2003. Chain of custody information was provided in Appendix C of the *Final Report on X-ray Fluorescence Field Study Of Selected Properties in Vicinity of Former USS Lead Refinery Facility, East Chicago, Indiana, USEPA Region 5, November 2003*.

- To add two additional Appendices. The additional appendices are as follows:

APPENDIX G - NITON Corporation's Service Report. This Appendix provides documentation of NITON's maintenance of the instrument used for XRF screening.

APPENDIX H - Copy of Field Notes, Vicinity of USS Lead, July 23, 2003 through August 21, 2003. All field notes from the project are included.

2.4 LABORATORY ANALYSIS

For each day that XRF screening of samples occurred, at least 1 sample was selected to be sent to the laboratory to be utilized for the project, American Analytical & Technical Services (AATS), for bulk sample confirmation analysis by USEPA SW-846 Method 6020. The XRF screening locations, and corresponding sample numbers, were identified in Table 3 of the *Final Report on X-ray Fluorescence Field Study Of Selected Properties in Vicinity of Former USS Lead Refinery Facility, East Chicago, Indiana, USEPA Region 5, November 2003*. Those XRF locations, and corresponding sample numbers are: X03/S03, X11/S04, X34/S07, X36/S12, X42/S14, X50/S17, X66/S20, X79/S23, and X83/S28. The results from the laboratory chemical analysis of these bulk samples, and the corresponding XRF results, are presented in Table 1.

3.2 XRF DATA QUALITY

After receipt of the laboratory data from analysis of bulk soil samples, USEPA examined which specific days the calibration of the Standard Reference Material (SRM) 2711 in the field indicate the XRF results exhibited low bias. This perhaps could then explain the samples for which the laboratory results would be expected to show greater concentrations of lead in the laboratory sample.

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The information summarized in Table 2 includes the percent difference between the XRF results for the SRM sample and the National Institute of Standards & Technology (NIST) certified value for lead (1162 ± 31 mg/kg), using the NIST certified results as the base for the percentage. As stated in the *Final Report on X-ray Fluorescence Field Study Of Selected Properties in Vicinity of Former USS Lead Refinery Facility, East Chicago, Indiana, USEPA Region 5, November 2003*, certain XRF screening results were anticipated to be biased low:

The next quality control check run was an XRF analysis of a Standard Reference Material (SRM) of known concentration, which was then compared to the certified values for the reference material. In this case the SRM used was SRM 2711, a Montana soil. The certified results for the SRM 2711 are contained in the SAP and in Appendix D to this report. The sample of the SRM was contained in a plastic cup specifically designed for use on the NITON soil testing platform. The SRM was acquired for use and prepared for the field effort by John Morris of USEPA's CRL. SRMs were run at the beginning and end of each day to ensure the instrument was working properly. The SRM check was considered acceptable if the XRF result for Pb plus the standard deviation of the result was within the low range of the certified standard. Otherwise, the XRF results should be expected to have a low bias. Results of all SRM quality control checks are shown in Appendices E & F.

Other possible explanations for the low bias associated with the XRF screening results were provided in TechLaw Inc.'s evaluation of the XRF results (TechLaw 2004). It was postulated that moisture, particle size and particle interface factors may have contributed to the low bias.

Clearly, all samples contained moisture and had to be air-dried prior to bagging a sample for analysis. Some were very wet (e.g. X79/S23 and X83/S28) and had to be air-dried for long periods prior to being suitable for XRF screening. Those samples in particular exhibited the highest percent difference between the XRF screening results and chemical analysis.

TechLaw Inc.'s evaluation of the XRF results (TechLaw 2004) also provides preliminary information on the analysis of lead in the fine and coarse fractions. The results from analysis of lead in the fine and coarse fractions are still under evaluation by USEPA and are not publicly available at this time. However, samples sent for analysis of lead in the fine versus the coarse fraction showed a greater concentration of lead in the sample of the fine fraction relative to the residual, greater than 150 micron, fraction. Based on this preliminary information, it was postulated that the XRF signal could be more strongly affected by the larger particles in the sample.

Further, TechLaw (TechLaw 2004) indicates that the particle size of the standard reference material (SRM) used to verify calibration of the instrument, SRM 2711, a fine-grained Montana

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soil, may have had smaller particle size than the soils examined in the study area and contributed to the low bias (TechLaw 2004). This is accurate, as the SRM 2711 particle size was clearly considerably smaller than the particle size of the field samples.

Table 3 summarizes the evaluation of the results from the confirmatory laboratory analysis, including the percent difference between the XRF results and the laboratory results. It is clear that the laboratory results are uniformly higher than the XRF results, with a percent difference ranging from 33.1% to as high as 161%. Based on the data, the XRF results are, on average, about 70% (69.6%) lower than the SW-846 Method 6020 results. The median percent difference between the XRF values and the bulk results is about 45% (45.3%).

Conclusions

Based on the evaluation of the results from the confirmatory laboratory analysis, the XRF data from samples where the XRF value exceeded the 400 parts per million (ppm) screening threshold are very likely to exhibit true values for lead exceeding 400 ppm. For the low end of the range (non detectable to 400 ppm), no samples were analyzed, allowing speculation as to the validity of XRF results for properties where XRF results are less than 400 ppm. The lowest XRF sample results sent for laboratory confirmatory analysis were samples X36/S12 and X66/S20, with XRF values of 549 ppm and 586 ppm, respectively. The laboratory results for those samples (see Table 2) were 31.5% and 59.5% higher than those values. It is therefore recommended that XRF results as low as 235 mg/kg for lead be viewed with caution as possibly being over the 400 ppm screening level.

4. REFERENCES

TechLaw, Inc. 2004. Evaluation of the EPA XRF Results for Lead. April 2004.

USEPA Region 5 2003. Final Report on X-Ray Fluorescence Field Study Of Selected Properties in Vicinity of Former USS Lead Refinery Facility, East Chicago, Indiana, USEPA Region 5, November 2003.

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Table 1. Results of XRF Analysis and Laboratory Analysis of Bulk Samples

Sample Date	SAMPLE ID/ XRF ID	Lead concentrations (Reported in milligrams/kilogram)	
		XRF Readings	Laboratory Analysis
7/23/03	S03/X03	1390	1850
7/24/03	S04/X11	1060	1540
7/29/03	S07/X24	827	1110
7/30/03	S12/X36	549	722
7/31/03	S14/X42	861	1230
8/5/03	S17/X50	736	1260
8/6/03	S20/X66	592	944
8/7/03	S23/X79	1624	4030
8/10/03	S28/X83	586	1530

XRF = X-Ray Fluorescence

Prepared after TechLaw, 2004

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Table 2. Evaluation of Results from Calibration of XRF Instrument with Standard Reference Material (SRM) 2711

Sample Date	XRF Calibration Results for Lead (milligrams/kilogram)					
	SRM 2711 AM	Calibration range met	% Difference * AM	SRM 2711 PM	Calibration range met	% Difference * PM
7/23/03	1040 + 47.2	Low	10.5	No Reading	Unknown	N/A
7/24/03	1089.6 + 44.5	✓	6.2	1069.6 + 47.9	Low	8.0
7/29/03	1109.6 + 48.5	✓	4.5	1089.6 + 48.6	✓	6.2
7/30/03	1069.6 + 47.3	Low	8.0	1129.6 + 48.8	✓	2.8
7/31/03	1120 + 39.3	✓	3.6	1060 + 45.6	Low	8.8
8/5/03	1089.6 + 46.6	✓	6.2	1000 + 45.3	Low	13.9
8/6/03	1100 + 43.5	✓	5.3	1100 + 44.4	✓	5.3
8/7/03	1100 + 48.5	✓	5.3	1120 + 38.6	✓	3.6
8/10/03	1069.6 + 45.7	Low	8.0	1049.6 + 44.5	Low	9.7

XRF = X-Ray Fluorescence

SRM 2711 = Standard Reference Material 2711, Montana Soil with Moderately Elevated Trace Element Concentrations

Each XRF result for SRM 2711 includes a concentration value and associated reading error. The SRM calibration data was reported in Appendix E to Final Report on X-ray Fluorescence Field Study Of Selected Properties in Vicinity of Former USS Lead Refinery Facility, East Chicago, Indiana, June 2004.

SRM 2711 Certified Value for Lead was reported as 1162 ± 31 mg/kg; XRF Results are expected to be biased low if XRF Screening Results plus error is less than 1131 mg/kg.

* % Difference is calculated as $[(\text{SRM Certified Value} - \text{XRF Reading}) / \text{SRM Certified Value} * 100\% = \% \text{ Difference}]$

AM and PM are reading time designations

Table 3. COMPARISON OF FIELD XRF AND LABORATORY RESULTS

Results of XRF Analysis and Laboratory Analysis of Bulk Samples				
Sample Date	SAMPLE ID/ XRF ID	Lead concentrations (Reported In milligrams/kilogram)		
		XRF Readings	Laboratory Analysis	% Difference *
7/23/03	S03/X03	1390	1850	33.1
7/24/03	S04/X11	1060	1540	45.3
7/29/03	S07/X24	827	1110	34.2
7/30/03	S12/X36	549	722	31.5
7/31/03	S14/X42	861	1230	42.9
8/5/03	S17/X50	736	1260	71.2
8/6/03	S20/X66	592	944	59.5
8/7/03	S23/X79	1624	4030	148.0
8/10/03	S28/X83	586	1530	161.0
Range				31.5 - 161.0
Mean				69.6
<p>XRF = X-Ray Fluorescence</p> <p>* % Difference is an absolute value calculated as $[(\text{Laboratory result} - \text{XRF reading}) / \text{XRF}] * 100\% = \% \text{ Difference}$</p> <p>% Difference as reported may be used to estimate corrected lead concentrations as follows $[(1 + \text{Difference}) * \text{XRF}] = \text{lead concentrations}$</p>				

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APPENDIX G

NITON CORPORATION'S SERVICE REPORT

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APPENDIX H

COPY OF FIELD NOTES

(Refer to File F.1 for original set of field notes)